THE ANALYST.

MAY, 1894.

DR. ARTHUR HILL HASSALL.

By the death of Dr. Hassall, which occurred at San Remo, there has been removed from our midst one to whom the public and all Public Analysts owe a debt of gratitude, though the period of his activity, during which he became widely known, lies so remote from the present time, that to many of the younger generation his personality represents little more than a name, and that one which is often pronounced with little respect.

Some months since, Dr. Hassall published an autobiography entitled "The Story of a Busy Life," which, though affording an insight into the life and character of this remarkable man, makes no reference to many matters on which information would have been acceptable; and which, to many who knew Dr. Hassall, would have been quite as interesting as those actually contained in the book. He has left his mark beneficially on a number of divisions of natural science, although he had to work under conditions of poverty and ill health, which would have proved an effectual bar to persons less energetic and mentally less active. Quite early in life he wrote a "History of the British Fresh Water Algæ"; in this many new species were described, and the work is, we believe, considered authoritative on the subject to this day. Soon after followed "The Microscopic Anatomy of the Human Body," which consisted chiefly of well-executed drawings; further, he wrote on "The Urine in Health and Disease," and he was one of the earliest to observe the occurrence of indican in the urine. Later still he founded the Royal National Hospital for Consumption and Diseases of the Chest at Ventnor, a hospital consisting of several blocks, and acknowledged to be a model of the Cottage Hospital type.

But it is mainly in connection with the adulteration of food that Dr. Hassall's claim to public recognition rests. As early as 1850 he applied the microscope to the examination of food, and though this at the present time may seem to be a mere matter of course, and one which requires no ingenuity, yet up to that date it had not occurred to any one to put this simple idea into actual practice. Dr. Hassall showed that it was easy to distinguish between coffee and chicory by means of the microscope at a time when this subject was a matter of Parliamentary investigation; and even after Hassall's discovery, the then Chancellor of the Exchequer, Sir Charles Wood, stated in the House of Commons: "I hold in my hand the report of the most distinguished chemists of the day, who state that neither by chemistry nor in any other way can the mixture of coffee with chicory be detected." This episode led to the institution, or rather the revival, by Mr. Wakley, at that time the editor of *The Lancet*, of the so-called "Analytical Sanitary Commission," which, in reality, was not

a commission at all, as it consisted solely of Dr. Hassall. The object of the so-called commission was to publish reports, partly for the public benefit, but mainly for the owners of *The Lancet*, on articles of food purchased in all the more important English towns, boldly stating the names of the vendors of the various articles, and the results of the examination of the same. These reports, which extended over a period of several years, and included the results of the examination of many thousands of samples of food, caused an immense sensation. They were mainly founded upon the examination under the microscope of those articles in which adulteration could be detected by that instrument; but it was gradually found that to this method of examination that of chemical analysis must be added. As Dr. Hassall had no chemical knowledge beyond that which had survived from his medical student days, he obtained the assistance of Dr. Letheby for that portion of the work. Ultimately the publication of *The Lancet* reports led to Parliamentary action being taken, and this resulted in legislation against the adulteration of food; the reports were subsequently collected by Dr. Hassall and published separately. We do not wish to do more than refer to the quarrel in the public press between Dr. Hassall and Dr. Letheby, both of whom claimed credit for the reports; nor to that between the editor of *The Lancet* and the member who formed the Sanitary Commission. After the publication of these reports, Dr. Hassall became for a time the chemical oracle of the public, and this upon the basis of his microscopic work, which part of his work was in every way excellent. To him public analysts owe most of their knowledge of the microscopic structure of food substances. His activity gave articulate form to the outcry against adulteration; he was truly the father of public analysis. In recognition of his great services he was elected the first vice-president of the Society of Public Analysts, but he never took any active interest in the formation or actual working of the society.

To the end of his life he remained what he essentially was, a microscopist, not a chemist, or an analyst in the modern sense of the word. His merits are none the less for that fact, and his name will be long and gratefully remembered by the members of the analytical profession.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

The usual Monthly Meeting of this Society was held on April 4th at the rooms of the Chemical Society, Burlington House. In the absence of the President, Mr. Otto Hehner took the chair.

The minutes of the last meeting were read and confirmed.

The following gentlemen were proposed for election as members: Messrs. Edward M. Chaplin, Thompson's Yard, Westgate, Wakefield; B. Henry Gerrans, 47, Aubert Park, Highbury, N.; William Marshall, 15, West Street, Rochdale; and B. H. Mumby, M.D., Portsmouth.

SOME FREQUENTLY-NEGLECTED ERRORS OF ANALYSIS.

By H. Droop Richmond.

In many commercial laboratories it is the practice for several experimenters to weigh on one balance and with one set of weights; and in more than one that I have known the weights are left on the pans when the weighings are finished, and the weight-box is left open. In laboratories where such is the practice, the standardizing of weights, and the daily adjustment of the balance to its zero point are also neglected.

I had occasion once to use a set of weights that had been used continually for some years in a busy laboratory, in which the standardizing of the weights was considered superfluous, and took the opportunity of comparing them to see to what extent errors might arise from this cause. The results were:

Nominal Weight.	Real Weight.	Nominal Weight.	Real Weight.
10′	Taken as Standard	·5	·4982
10″	9·9997	·2	·1993
5	4·9990	·1′	·0971
2	1·9991	·1″	·0982
1′	·9987	·05	·0485
1″	·9989	·02	·0191
1‴	1·0008	·01′	·0092
		·01″	·0089

As an example of the error which may occur, I quote the analysis of a commercial sulphate of copper. This is an actual example obtained with other weights; I have supposed, for purposes of illustration, that the above weights were used:

Tare of watch glass	4·3370	
Watch glass and sample ...	5·3387	Supposing 1‴ had been added
	1·0017	the real weight is 1·0025
Tare of Platinum	13·0032	
Platinum and Copper	13·2373	Supposing ·2, ·02, and ·01′ had
	·2341	been added the real weight is ·2317

	Apparent.	Real.
Percentage: Cu	23·37	23·11
$CuSO_4 \cdot 5OH_2$ ...	91·83	90·82

I have here chosen a striking case for purposes of illustration, but it is one that may occur in many laboratories, and may lie in opposite directions with different analysts; this possibly is a partial explanation of the differences between chemists in commercial analysis. I have found that two or three hours is sufficient for the comparison of a set of weights, and would recommend Gauss' method of reversal (*cf.* Miller, *Phil. Trans.*, 1856, iii.) as the most useful method of determining them.

In the majority of balances that I have used the zero point has varied with the load; thus in a long-beam Oertling I found:

Load in each Pan.	Zero Point in Milligrammes.
none	0·0
1 gramme.	+0·3
2 ,,	+0·6
5 ,,	+1·0
10 ,,	+1·2
20 ,,	+1·7
50 ,,	+3·6

By the ordinary method of weighing there would be ·03 per cent. difference in the apparent value of a 1 gramme weight, according as it were weighed with no other load or with a load of 50 grammes. This would be of no practical importance in ordinary work, but it illustrates the absurdity of stating results to the third place of decimals per cent., which I have seen on commercial certificates of analysis.

Neglect of correction of weighings to a vacuum is invariably practised in commercial analysis, yet it may affect the fourth place of decimals in density determinations of strong sulphuric acid, and may cause errors of nearly 0·1 per cent. in normal solutions, if barium sulphate, or silver chloride, or bromide be weighed.

That graduated instruments are frequently incorrect is universally known, but in many laboratories they are used as sold without being checked.

It is a less well-known fact, though a committee of the British Association has reported on this, that blowing out pipettes is less accurate than simply allowing them to drain, and then touching the end of the pipette against the side of the vessel in which it has been allowed to drain. It must also be remembered that a pipette which delivers 10 c.c. of water will not deliver the same volume of sulphuric acid or milk or of any fluid of differing viscosity; two 10-c.c. pipettes, each correct for water, may not deliver the same volume of sulphuric acid, and errors in oil analysis by the Maumené method may thus arise.

Turning to the errors of standard solutions, the expansion by heat is a cause of error; thus, with normal sulphuric acid, calling the value 15° 100, the following values at various temperatures are found:

Temperature.	Value.
10°	100·12
15°	100·00
20°	99·85
25°	99·69
30°	99·52

As the extremes in the table may be taken to represent the temperatures attained in a laboratory in summer and winter, it follows that a difference of 0·6 per cent. may be due to the neglect of expansion of a standard solution.

Another error in alkalimetric titrations may occur from the standardization of baryta solution on normal sulphuric acid; I have already referred to this (ANALYST xvi., 166), but as since then Richards has redetermined the atomic weight of barium, I have corrected my figures for this.

Thus the standard acid used contained 5·08° per cent. of H_2SO_4 (using 137·5 as the atomic weight of barium and correcting the weighings to a vacuum standard) instead of 5·104 per cent. The percentage deduced from Pickering's tables was found to be, on fully correcting the density determinations, 5·093 per cent., the corrected densities being

at $\frac{1}{4°}$ 1·00381

and $\frac{20°}{4°}$ 1·03218.

The following determinations of strength of the baryta solution were found at 25° :

14·14 c.c.	gave ·1705 grs.	$BaSO_4$	= ·1041	Normal
39·48 ,,	,, ·4790 ,,	,,	= ·1039	,,
29·48 ,,	,, ·3588 ,,	,,	= ·1042	,,
Titrated with $\frac{N}{10}$ Hydrochloric Acid			= ·1040	,,
Titrated with Oxalic Acid			= ·1042	,,
	Mean ...	...	·1041	,,

Titrated with the Sulphuric Acid referred to above it was

		·10301	Normal
		·10315	,,
			,,
Mean	... ...	·10308	,,

It is seen that titration with sulphuric acid shows the baryta solution to be 1 per cent weaker than it really is, and this is no doubt due to the carrying down of barium hydroxide by the barium sulphate.

I may mention also that a standard silver solution has the same property, and that in Mohr's method of chlorine estimation, the value of the silver solution as estimated from the silver contained differs from that obtained by titrating pure sodium chloride by fully 1 per cent. (see ANALYST, xiv., 229). The work of Stas has explained this. The influence of carbon dioxide in solutions of acid has been lately discussed.

With one more fact which is not widely recognised, I bring this somewhat rambling paper to a close, Platinum is permeable at a red-heat by the reducing gases of a Bunsen flame, and precipitates such as copper oxide and manganese dioxide may be somewhat reduced, and the copper oxide may contain less oxygen than CuO, and the manganese may be weighed as a lower oxide than Mn_3O_4. This may be avoided by the use of the muffle.

DISCUSSION.

Mr. Hehner thought the society would feel obliged to Mr. Richmond for calling attention to facts with which all analysts were supposed to be acquainted. It was highly desirable to test weights occasionally.

Dr. Walter J. Sykes mentioned that properly graduated instruments could be obtained from some firms on the Continent at prices a little higher than those charged for the ordinary ones in England.

Mr. Arthur Ling said it was desirable that the measuring-vessels used for volumetric analysis by one observer, or in any given laboratory, should be verified by means of one set of weights, because sets of weights often differed in standard. With regard to the measurement of liquids by pipettes, one of the best plans was that advocated in Thorpe's Dictionary, viz., to allow the contents of the instrument to drain, and then touch the surface of the liquid once with the point. The most accurate pipettes, however, were probably those having two marks, one above and the other below the bulb.

Dr. Morgan said that the error in regard to weights was very much reduced if one operator followed his work right through with one set of weights. It was not desirable to have sets of weights by various makers in a laboratory.

Mr. Hehner said that he had never found a serious error in the calibration of pipettes. Standard solutions were mostly exceedingly dilute, compared with the strong sulphuric acid to which Mr. Richmond referred. No doubt for very accurate work, where it was necessary to reduce weights to vacuum value, it was advisable to check everything, but, for ordinary work, errors due to weights which had become a little worn were more or less compensated by other errors. He did not wish to be understood to say that, in his opinion, it was desirable to use very highly-worn weights, but it must be within the experience of most chemists that good weights which had become black and tarnished, when tested were found to be still accurate.

Mr. Richmond said that he wished to draw attention to the importance of accuracy in detail; an analyst who took the small precautions of standardizing weights, etc., would undoubtedly furnish to his clients more reliable results than if he left such things to chance.

The following papers were also read: "The Analysis of Waters for Minute Quantities of Poisonous Metals," by E. Russell Budden; "Note on the Analysis of Phosphor Tin," by Frank L. Teed, Ph.D.; "Note on Lemon and Orange Peel," by E. G. Clayton. The publication of these is unavoidably held over.

NOTES FROM THE LABORATORY OF THE ROYAL AGRICULTURAL SOCIETY OF ENGLAND.

By J. Augustus Voelcker, Ph.D., B.Sc.

(*Read at the Meeting March 7th*, 1894.)

I. The Occurrence of a Poisonous Leguminous Seed in Indian Peas.

"Indian peas" is a vague term applied almost indiscriminately to a number of different leguminous seeds imported from India, and used for feeding purposes. The principal pulses thus used are the following: Gram (*Cicer arietinum*); *arhar*, or *dâl* (*Cajanus indicus*); the common pea (*Pisum sativum*); and the field pea (*Pisum arvense*), the last two being generally known as *mattar*.

Previous to my visit to India in 1890, I had not unfrequently come across instances where injury to stock, and even death, had been attributed to the use of

some mixed feeding-cake or other; but, on examining the cake, I had been unable to find anything to justify the suspicion. It is well known to agricultural chemists that one of the dangers of mixed feeding-cakes consists in their being made of materials originally unsound; but after the constituents have been cooked and pressed together into cake, it has been impossible to know what they were like at first. Of late years, too, agricultural chemists have been careful to search for the possible presence, in mixed cakes, of seeds which are known to have poisonous properties. I might refer here to a paper read before this society by my late assistant, Dr. Leather, on the detection of castor-oil bean in feeding-cakes.

In several instances brought to my notice before I went to India, I found that farmers had been tempted by the low price of "Indian peas" to purchase these instead of home-grown peas, and had experienced considerable trouble with the stock to which these peas had been given; in most cases symptoms of paralysis were shown. I was unable at the time to account for this, but so certain was I that the complaints had some real basis that I was led to regard "Indian peas" with considerable suspicion. Accordingly, when I went to India in 1890, I made a special point of inquiring into this matter; and I believe that I have arrived at the right solution. Since my return in 1891 I have watched the subject further, and I have within quite recent times had three cases brought to my notice in which harm has been attributed to the use of mixed feeding-cakes or meals, my subsequent examination showing there to be present in them the seed of a vetchling called *Lathyrus sativus*. This was the same seed that I met with in India, and which is known there to possess poisonous qualities. A specimen of the seed is here shown. It has a very unusual and characteristic shape, being ovate, or wedge-shaped, flattened on two sides, and having a very mottled or "marbled" appearance, and also a continuous thin black line running over about two-thirds of the circumference of the seed. The colour is variable, generally grayish, but it ranges from yellowish-brown to nearly black, and all seeds show the marbling to a more or less extent. Its size is about that of our English pea, but the shape is very different, and the seed is not rounded at all.

Lathyrus sativus, Linn., or *khesâri* pulse, is a crop grown principally in Northern India, and mainly as a fodder crop. The plant is a procumbent or climbing herb, the crop looking like a short one of vetches. It is grown on poor land that will raise no other kind of pulse, and generally after much rain has fallen. Duthie and Fuller, in their book "Field and Garden Crops of the North-West Provinces," speak of it as a coarse kind of pea, notorious for its effect in producing paralysis if eaten in excess; and they mention that the widespread occurrence of paralysis in Sindh, after a season of extensive inundations, in which *khesâri* was grown on an exceptionally large scale, was due to the consumption of it, and also that cases of paralysis are far from uncommon in villages where *khesâri* forms an important item of diet. A case is cited where, at the military station of Almora, some cases of paralysis were traced to the fraudulent admixture of *khesâri* with the gram supplied for the use of the troops.

Colonel Sleeman also writes upon the ill effects of a large consumption of the grain in the province of Oudh in the years 1831 and 1832. Many of the people lost the use of their lower limbs entirely, and, when once attacked, never recovered completely. He adds that "it is the prevailing opinion of the natives throughout

the country that both horses and bullocks which have been much fed upon *khesári* are liable to lose the use of their limbs; but if the poisonous qualities abound more in the grain than in the stalk or the leaves, man, who eats nothing but the grain, must be more liable to suffer from the use of this food than beasts which eat it merely as they eat grass or hay." The fact that the crop is principally used as a fodder crop is the probable explanation of the poisonous properties of the grain, when used as food for cattle, not being so well known among us.

In Dr. George Watt's "Dictionary of the Economic Products of India," it is pointed out that "an analysis by Astier has revealed the presence in the grain of a volatile liquid alkaloid, probably produced by some proteid ferment, which exhibits the toxic effects of the seeds, and the action of which is destroyed by heat." Numerous cases quoted in Dr. Watt's "Dictionary" leave no doubt as to the poisonous effects produced by the too free use of the grain. In horses, paralysis of the hinder extremities takes place, as also an affection of the larynx, ending in asphyxia and death. To the particular condition produced by this over-feeding, the name "lathyrismus" has been given. Dr. Watt refers to the importance of the fact that the alkaloid recently isolated is volatile, and that while in grain cooked at a high temperature it may be driven off, yet when the seed is cooked at a low temperature, or when used as grain for cattle, it may retain its poisonous properties. In this way the somewhat uncertain and variable effects of the grain may be explained.

I saw the crop growing in India myself, and made inquiries about it on the spot. These were quite in confirmation of the remarks made above, and I obtained also there the specimens of the seed which I now produce.

In the first of the three cases lately brought to my notice, in which poisonous results were observed from the use of this seed, the farmer complained that he had lost a quantity of beasts and sheep that had been eating a certain mixed feeding-cake. In the cake I found present seeds of the vetchling *Lathyrus sativus;* but, inasmuch as it transpired in the course of the inquiry that some of the cattle had been eating acorns, it was impossible to attribute the loss directly to the presence of the vetchling, and to say whether death resulted from it or from acorn poisoning. In a second instance I was informed by a firm of cake-makers, whom I had previously warned against buying "Indian peas," that they had inadvertently bought some, and had used them in the manufacture of a small lot of mixed cake. Within two days of sending out the delivery they had a complaint about it, and at once recalled the cake when they found the kind of peas that had been used in the manufacture. On examining the peas used, I found the *Lathyrus sativus* present.

Not above a fortnight ago I received a letter from a veterinary surgeon, stating that a farmer in his neighbourhood had, previous to his visit, lost two milch cows, and that on his arrival he had found four other cows unwell. "The most conspicuous symptoms," he added, "were paralysis." The cows had been feeding on a mixed meal got from a dealer in the neighbourhood, and a sample was sent up to me for analysis. I have here some of it, and a short examination showed me that there were present in it seeds of the *Lathyrus sativus*.

I was not able to ascertain more as to the origin of the meal than that it contained a portion of pea-meal, but I could not hear where the peas came from.

Lastly, I have at the present time a sample of mixed feeding-cake to examine, in which I have noticed whole seeds of the *Lathyrus sativus* present.

I have little doubt that in several cases where harm has been attributed to the use of mixed feeding-cakes or meals, or to that of Indian peas, the cause might have been found in the presence of this poisonous seed *Lathyrus sativus*. It is not distinguishable from other pulses by any peculiar microscopical appearances, or by any difference of its starch; but the marbling is very characteristic, as is also the shape of the seed, and the thin black line running nearly round the seed. It is well to point out that seeds of a greenish colour, and having a similar marbling to *Lathyrus sativus*, frequently occur among the ordinary varieties of pea, *Pisum sativum*, or *Pisum arvense*, and must not be confounded with it. The *Lathyrus* seed is flattened in shape, and not rounded like the seeds of *Pisum sativum* and *Pisum arvense*.

Discussion.

The Chairman (Mr. A. H. Allen) said that Dr. Voelcker had undoubtedly brought forward a very important subject. Analysts ought to be very much indebted to anyone who would point out these minute differences between innocent seeds and poisonous species.

Dr. Bernard Dyer said that Dr. Voelcker was the first to call his attention to this matter. He had certainly met with several cases in which very virulent poisoning had followed the eating of peas coming from the East. He remembered a case of Persian peas coming into the market. A miller ground them into meal, which he sold to several customers, and with which he commenced feeding his own pigs. His pigs died promptly, and he telegraphed to his various customers, but in several cases their pigs were also dead or dying. Not long ago he had had brought to him some water by a firm of wharfingers to see if anything poisonous had been put into it, because their horses had died and others had been unwell, and it was thought that the cisterns had been poisoned. There was nothing abnormal in the water. He found, however, they had been feeding the horses on Indian peas. Unfortunately they had none of the peas left, so that it was impossible to find out whether any of them were poisonous; but the dates between which they were given to the horses coincided, he understood, with the time during which the illness and death of the horses took place.

Dr. Dupré said that the fact that the boiling sometimes rendered the peas innocuous, did not, in his opinion, point to a volatile alkaloid being present, as one could scarcely imagine that a short heating would expel all the volatile alkali. The effects observed rather indicated poisoning by organisms. In a case of poisoning which occurred some years ago, and which was due to the eating of bread-and-butter pudding, it was found that the puddings were made from the refuse bread of a coffee-house, some of which had become mouldy. One of the puddings which had been well baked proved harmless, the second was imperfectly cooked, and was poisonous. That the poisonous character of these peas might be due to the production of poisons by organisms, seemed to him worthy of consideration.

Mr. M. A. Adams thought it was a remarkable thing that of an alkaloid it should

have the property of producing paralysis. It had struck him that the poisonous substance must be rather of the nature of an albumose than an alkaloid. The fact of the seed containing a paralytic poison suggested to his mind that it came within the class of albumose substances. Until something was done, chemically, to prove the contrary, he was disposed to the idea that probably the poison was foreign to the seed.

Mr. John Hughes asked Dr. Voelcker if he had made any estimation of the amount of the poisonous seeds present in the feeding cake.

Dr. Frank L. Teed believed that Dr. Voelcker had commenced by saying that these poisonous seeds were grown as a fodder crop. The matter therefore seemed to him to require some sort of explanation, because it seemed very strange that a crop which was known to be poisonous should be grown. He (Dr. Teed) inferred that all that Dr. Voelcker had in any way demonstrated was that Indian peas were sometimes dangerous. He thought the explanation suggested by Dr. Dupré would have great weight, and that explanation ought to be thoroughly investigated before the seed of a non-poisonous plant received the condemnation which Dr. Voelcker wished to place on it from apparently insufficient evidence. He wished to know if Dr. Voelcker had any direct evidence of the poisonous action of these seeds.

Dr. Dyer anticipated Dr. Voelcker's reply by pointing out that, in our own country, and more especially in Ireland, a fodder crop very largely used for horses was grown, viz., furze or gorse. Although this was a very favourite fodder for horses, the seed, nevertheless, was virulently poisonous, containing the alkaloid ulexine, similar in its toxicological action to the laburnum poison.

Mr. Otto Hehner wished to ask Dr. Voelcker one question, from an analytical point of view. Dr. Voelcker had informed the Society that there was no microscopic difference in the structure of these seeds, and he seemed to recognise them solely by the form, the shape, and the wrinkling or the corrugations. If these seeds were crushed or made into meal, which was a very probable contingency, how would the analyst be able to deal with the matter?

Mr. H. Droop Richmond drew attention to the fact that Dr. Vaughan some time ago investigated some cases of ice-cream poisoning in the United States. It was found that a poisonous alkaloid had been developed in the ice cream; this was isolated by Dr. Vaughan, and named "tyrotoxicon." The identity of tyrotoxicon with diazobenzene butyrate had since been established.

Mr. F. H. Perry Coste said that, in connection with Mr. Richmond's view, danger from poison could be got rid of by roasting in the case of acorns; which had been, he believed, cooked and eaten by various barbarous nations, who presumably would not eat them if they were poisonous. He did not see that there was any *a priori* improbability of an alkaloid being present in this case. Besides, as Dr. Dyer had mentioned, an alkaloid had been isolated from *Ulex*. It was also known that the seeds of the Laburnum were exceedingly poisonous, and had sometimes been eaten by children with fatal results.

Dr. Voelcker quite agreed that only imperfect knowledge of the subject at

present existed. It was probable that the poison was either an alkaloid or an albuminoid ferment. He had not estimated the actual quantities of the bean present in different foods; the difficulty was to judge from the amount of the husk how much of the original seed had been present. In regard to what Dr. Teed had said, from an agricultural point of view no explanation was needed. He would cite the case of a mustard crop, which was injurious as regards the seed, but quite harmless when used as a green crop. Cases of paralysis were frequent in the districts where the *Lathyrus* was cultivated. It was essentially a matter of the quantity present; a small quantity might not produce paralysis.

II. Analyses of Waters from Wells in close proximity to Churchyards.

It is not always that one can obtain definite information regarding the surroundings of wells, the water from which one is called upon to analyze, and when the particulars are forthcoming the chief points brought out by the analyses may be found useful for future guidance. Having recently analyzed three waters which I reported on adversely, and then finding that the wells were in close proximity to churchyards, I thought it might not be devoid of interest if I gave the analyses. The results were as follows:

	I. Grains per Gall.	II. Grains per Gall.	III. Grains per Gall.
Total solids...	23·52	38·36	69·99
Oxygen absorbed	·039	·066	·055
Chlorine	2·28	4·02	6·78
equal to Sodium chloride... ...	3·65	6·57	11·07
Nitric Acid	3·67	5·13	12·87
Free Ammonia	·006	·0005	·0005
Albuminoid Ammonia	·006	·013	·013
Phosphoric Acid	·29	·34	·52
equal to Phosphate of Lime ...	·59	·74	1·13

The wells from which waters I. and II. came were from the same place, and only about 80 yards apart. Water No. I. was not clear, was somewhat yellow-coloured, and had some amount of suspended matter. It left on evaporation to dryness a yellow residue. As the analysis showed, it was a water clearly open to grave suspicion. Water No. II. was free from any suspended matter or deposit, but was of a bright yellow colour. The chlorides and nitrates, as well as the total solids, were considerably more than in No. I., and the water was clearly badly polluted. On inquiry I found that both wells were within 25 to 30 feet of a churchyard, and that in a family which had drunk of the No. II. water, one, and it was believed two, members had died within the past three weeks of pronounced diphtheria. On inquiring further, so as to ascertain, if possible, the cause of the differences shown in the two analyses, I was told that while well No. II. was only 50 feet distant from *several* recent burials, well No. I. was 140 feet from *one* recent burial. The subsoil was hard gravel.

In the case of water No. III. the results were even more alarming. The water was of a yellowish colour, but was quite clear, and free from deposit. I was informed that the well was 23 feet deep, there being 8 feet of water in it; that a churchyard was only 100 feet distant, and a closet-drain 80 feet off. There had been illness here also from drinking the water. This water contained a considerable quantity of magnesia. Besides the high results in chlorides and nitrates shown in these analyses, it is be noticeable that in two out of the three cases a mere trace only of free ammonia was shown; and these instances prove that the significance of nitrates, even when a minute quantity only of ammonia is present, is very great, and ought never to be overlooked. I thought it desirable also to determine the phosphoric acid in the samples, and the results given are very high, as one might expect in the case of polluted water-supplies.

Discussion.

Mr. Alfred H. Allen remarked that those analysts who were in the habit of making daily examinations of water for sanitary purposes were perfectly aware of the presence of nitrates in impure waters of this kind, although, unfortunately, their presence and indications were ignored by certain much-quoted authorities, with very great harm to the country generally. It was not at all unusual in his experience to find waters almost destitute of free ammonia, and yet containing large amounts of nitrates and chlorides. As to the phosphoric acid, he found that it was present very frequently in traces. He had, however, ceased to rely on it as an indication of contamination of the water, but he had never found anything like the amount stated by Dr. Voelcker. No doubt the proximity of the churchyard might account for these amounts.

Mr. M. A. Adams wished to ask two questions—first, as to the physical relation between the wells and the churchyard and the kind of well; and, secondly, as to the nature of the disease which was attributed to the drinking of the waters. He might say that from his experience of cases of diphtheria, he considered that this was not a water-borne disease. He would like Dr. Voelcker to tell him whether there had been any other disease in any way traceable to the waters. If so, it was important to know how the association between the waters and the disease was traced.

Dr. Teed asked Dr. Voelcker whether there was any other source of contamination in these waters; as from his experience one of them certainly appeared to be contaminated with sewage, and had not the characteristics of a cemetery water. Cemetery effluents were very rich in organic matter, but not in chlorine.

Mr. Benedict Kitto asked whether the waters were taken anywhere near the sea-coast, because he very frequently had such waters. Though they contained very high amounts of chlorides indeed, he passed them as quite safe for drinking purposes.

Mr. H. Droop Richmond asked Dr. Voelcker if he knew whether the fields in the vicinity had been treated with artificial fertilizers, because in one case he found a water containing a very large amount of phosphoric acid and nitrates, traceable to artificial fertilizers used on the fields, and the water was proved not to be contaminated except in this way.

Dr. Voelcker, in reply, said that on the medical side of the question he could say nothing. He had received the waters to examine, and he had reported on them. Subsequently, as a matter of interest, he had inquired whether there was anything to account for the state of things found. As regards the position of the wells, the first two were in Worcestershire, and the other at Tewkesbury, in Somersetshire. The soil in the first two cases was a sandy gravel, but he had no information as to the soil from which the last came. It was evidently a very much harder water, and there was a good deal of sulphate of lime in it, such as might be found in Somersetshire. In reply to Mr. Richmond, he did not know how a distinction could be drawn between the bones that lay in a churchyard and bones that might have been used as manure on a field. They both came under the category of manure, so that their effects might be alike, whether proceeding from the field or from the churchyard.

The Analysis of Rubber Goods. C. A. Lobry de Bruyn and **F. H. van Leeut.** (*Chem. Zeit.*, 1894, xviii., 309-312.)—The authors review various methods hitherto in vogue and dismiss many as illusory, notably such as assume that a rubber of low specific gravity, or one containing but little mineral matter, is necessarily of good quality—an invalid assumption in view of the fact that rubber containing surrogate may answer tests of this kind. An empirical method adopted by the Admiralty and capable of yielding useful data, consists in heating the rubber goods to be tested, dry to 135° C., and, with water in a sealed tube, to 170° C. The test discriminates between pure rubber of good quality, and that which is genuine but of poor quality, from the presence in it of an undue proportion of rubber resin, and also that which is adulterated with surrogate. Vulcanized Para rubber, alone and mixed with mineral ingredients (*e.g.*, zinc oxide), withstands exposure to the temperatures quoted for 2-4 hours without perceptible deterioration. Inferior goods are more or less damaged by the treatment, becoming hard and brittle and losing their elasticity, or swelling and fusing. The dry-heating test is carried out so that the loss of weight of the rubber is determined at the same time. Good vulcanized rubber, cut into thin shreds, loses 2 per cent. of its weight as a maximum at 135° C., the loss usually being less than 1·5 per cent. Thus a specimen of pure Para rubber lost 0·15 per cent. after one hour, and 0·25 per cent. after three hours; a sample from Mozambique 0·2 and 1·3 per cent. at the same periods, while the corresponding figures for a Borneo rubber were 1·7 and 3 per cent. The percentages of rubber resin in these three samples were 5, 6·5 and 36 per cent. respectively.

In judging rubber from its loss of weight it must be remembered that many common rubbers containing surrogate give low figures like genuine rubber containing a moderate proportion of rubber resin.

In applying Henriques' method for the detection of saponifiable constituents in rubber by extraction with alcoholic alkali (The Analyst, xviii. 13), the existence of resinous substances naturally present in rubber and soluble in the same menstruum must not be forgotten. As much as 40 per cent. of the total rubber substance may consist of bodies of this class, and their nature varies to some extent with the origin of the rubber of which they form part. The authors, while acknowledging the value

of Henriques' work, state that in their opinion the methods laid down by him are too lengthy for use when a rapid decision concerning the quality of a number o samples of rubber has to be arrived at, and append the results of some simple tests of a number of rubber articles of different sorts (*v. i.*). They note that when detailing the composition of rubber which is to be manufactured according to a definite specification, zinc oxide may be conveniently adopted as the mineral constituent, unless the rubber is to come into contact with acid liquids, when barium sulphate is a more suitable "filling" material. Zinc oxide is especially objectionable in laboratory corks that are used in such processes as the determination of an acetyl group in an ester by digestion for some six hours with $\frac{N}{10}$ sulphuric acid, low results being obtained. Chalk, which was present in a sample of rubber tubing to the extent of 70 per cent., is equally obnoxious. In carrying out the tests on the samples quoted below, the ash was determined by gentle ignition without special oxidation and carbonation—practices sometimes resorted to—and results of fair constancy, sufficient for judging the quality of the goods, were obtained. The determination of the percentage of matter soluble in alcoholic soda was carried out in the manner prescribed by Henriques, and in the case of rubber containing mineral constituents was calculated on the ash-free material. The estimation of the loss on heating to 135° C. for two hours was usually performed only on rubber free from ash, and was made on pieces of rubber not more than 1 mm. thick, placed in a drying-oven previously heated to the required temperature. The test, consisting in heating the rubber with water under pressure, was carried out by placing the samples (not subdivided) in a strong copper or iron tube closed with a screw and capable of withstanding a pressure of at least ten atmospheres, about half or two-thirds full of water, raising the tube and its contents to 170° C., and maintaining this temperature for four hours.

In the selected list given below the samples are arranged in the order of their content of ash; those for which no ash is recorded were practically ash-free, *i.e.*, contained not more than 1 per cent. The specimens marked with an asterisk withstood both heat tests:

No.	Description.	Ash Per Cent.	Percentage soluble in 6 per cent. Alcoholic Soda, calculated on the Rubber Free from Ash.
1.*	Vulcanized Para rubber, 6·4% S.	—	5
2.*	Vulcanized Mozambique rubber, 5·5% S.	—	6·5
3.	Vulcanized Borneo rubber, 8·3% S.	—	36
9.	Old laboratory cork	4·5	7
10.*	Tubing ("patent" rubber)	—	4·9
15.*	Black rubber ring	6·5	0·7
17.	Rubber disc	15·5	9
18.	Rubber ring	15·5	23·3
23.	Red tubing	24·5	12
25.	Rubber mat	44	50
26.*	Rubber cork (French make)	44·5	11·9
27.	Rubber packing	45	23
36.*	Rubber valve	51·5	4
60.	Rubber plate	71	21·2

Certain of these samples were examined for their loss on heating to 135° C.:

No.	Loss after One Hour.	Two Hours.	Three Hours.
1.	0·01%	— %	0·25%
2.	0·02	—	1·3
3.	1·7	—	3
15.	1·1	2·5	—
18.	—	3	—
25.	—	—	4

It is evident from the foregoing figures that a genuine rubber containing little rubber resin but much ash resists destructive influences better than an adulterated or resinous sample (yielding a large quantity of extractive matter on digestion with caustic soda), even though the latter be fairly free from ash.

White and brown surrogates lose 2 to 2·5 per cent. on heating to 135° C., but do not fuse at this temperature, and indeed are but little altered; when subjected to the hot-water test, however, they fuse, darken, and decompose with the production of evil-smelling sulphur compounds. According to the authors' experience, rubber adulterated with surrogate does not stand the hot-water test. As stated above, it is generally true that samples yielding only small quantities of extract on treatment with alcoholic soda withstand the heat tests, but the rule is not universal. The age of the rubber is a factor that cannot be overlooked, as it is well known that even good rubber after years of use becomes much deteriorated. The authors call attention to the fact that the manufacture of rubber goods specially adapted for different laboratory purposes is very desirable. Thus corks and tubing containing much insoluble mineral matter (*e.g.*, $BaSO_4$) would be useful for purposes where contact with corrosive liquids and gases is inevitable, while all other laboratory rubber articles should be of pure ash-free rubber. B. B.

The Analysis of Rubber Goods. R. Henriques. (*Chem. Zeit.*, 1894, xviii., 411, 412, and 442-444.)—The author has continued his endeavours to arrive at reliable general methods for the analysis of rubber goods; these methods have been already abstracted into the ANALYST. In his former work he elaborated processes for the separation and estimation of fatty oils and oil surrogates in rubber, and now he has been able to check the accuracy of these methods by the analysis of rubber goods of known composition. Three samples—one containing brown surrogate, another white surrogate, and the third rape-oil—were prepared by a friendly manufacturer and analyzed by the author, with the results given below.

Before detailing the result of this work, it may be remarked that a minor improvement in the method originally used has been made. The fundamental separation of surrogate from rubber depends on the saponification of the former, and it is found that the rubber obstinately retains a certain amount of alkali, necessitating a correction in the estimation of the true rubber substance by determining both the total increase of ash and the sulphur contained therein. This complication can be in great measure avoided by boiling out the rubber, after treatment with alcoholic alkali to remove surrogate, with dilute hydrochloric acid. Not quite all the alkali is thus removed, some 2 to 3 per cent. remaining; but the quantity of sulphur retained

is negligible, and the difference between the original ash and that obtained after the successive treatments with alkali and acid, added to the apparent value for the soluble surrogate, gives the true amount thus extracted. This modification, involving the use of an acid solvent, is applicable in the case of rubber containing soluble inorganic constituents, as these are first removed by treatment with acid before the determination of the surrogate is begun. The following data are required in the determination of the composition of rubber goods containing surrogate (or oil) rubber and sulphur: (*a*) Total sulphur, (*b*) total ash, (*c*) residue after extraction with alkali, (*d*) sulphur in this residue (calculated on the original rubber), (*e*) ash in this residue (calculated on the original rubber), (*f*) sulphur in the extracted fatty acids. For the determination of *c*, 1·5 to 2 grammes of the substance should be boiled for 2 to 3 hours with alcoholic soda, the extract being poured off and the treatment repeated. A mean correction of 2·5 per cent. is made for the amount of true rubber dissolved, this value being the average of ten determinations with various genuine samples of rubber, which gave figures ranging from 0·9 to 3·5 per cent. The determination of the other data (*a*, *b*, etc.) is performed according to the prescription already detailed by the author in his former papers. Then, calling the percentage of pure rubber x, and that of the fatty acids y, we have:

$$\frac{2{\cdot}5x}{100} + y = 100 - c + (e - b) - (a - d)$$

and

$$x + y = 100 - (a + b)$$

solving which we obtain:

$$x = \frac{100}{97{\cdot}5}(c - d - e)$$

$$y = 100 - (a + b + x).$$

The value of f is obtained from the facts, arrived at in an earlier part of the investigation, that the sulphur content of white surrogate is identical with that of its fatty acids, and that the sulphur content of brown surrogate is on the average 1·5 per cent. higher than that of its fatty acids. Finally, the sulphur used for vulcanizing is obtained as the difference between the total sulphur and that contained in the surrogate. The analyses of the three samples mentioned above were conducted on these lines, the figures obtained being:

	I. Rubber + Brown Surrogate.	II. Rubber + White Surrogate.	III. Rubber + Oil.
(*a*)	19·46	11·38	21·55
(*b*)	0·72	0·40	0·40
(*c*)	68·37	67·68	63·40
(*d*)	2·64	1·51	3·06
(*e*)	2·70	2·81	3·17
(*f*)	16·60	9·27	3·20
Chlorine.	absent.	much present.	absent.

The composition of the three samples deduced from these figures and arrived at synthetically are compared below:

I. *Rubber + Brown Surrogate.*

	Analysis.	Synthesis.
Sulphur	16·13 per cent.	16·4 per cent.
Brown surrogate ...	18·50 ,,	19·3 ,,
Rubber	64·65 ,,	64·3 ,,
Ash	0·73 ,,	—

II. *Rubber + White Surrogate.*

	Analysis.	Synthesis.
Sulphur	9·19 per cent.	9·6 per cent.
White surrogate ...	25·43 ,,	25·8 ,,
Rubber	64·98 ,,	64·6 ,,
Ash	0·40 ,,	—

III. *Rubber + Oil.*

	Analysis.	Synthesis.
Sulphur	21·55 per cent.	23·5 per cent.
Oil	20·42 ,,	17·7 ,,
Rubber	57·63 ,,	58·8 ,,
Ash	0·40 ,,	—

It is evident from the foregoing figures that the concordance between the calculated and ascertained results is good in the case of Samples I. and II. containing white and brown surrogate, but that it is less close for the mixture of rubber and oil (No. III.). The author attributes this divergence less to error of analyses than to the character of the sample analysed. Although of normal aspect, it was thoroughly porous, as if an evolution of gas had occurred during vulcanization (causing loss of sulphur as H_2S). It is also an acknowledged fact that fatty oils strongly attack rubber at high temperatures, and resolve it into products soluble in alkali. This accounts for the presence of sulphur in the fatty acids in not inconsiderable amount (3·20 per cent.), although the oil originally used was necessarily free from sulphur. The discrepancies thus occasioned are not of much practical importance, as rubber containing considerable quantities of fatty oil is not met with in commerce, the only sample which the author has examined being an unvulcanized mixture for the preparation of hard rubber, the oil in which would be converted into surrogate in the act of vulcanization by the excess of sulphur present.

A contribution to existing methods for separating rubber from fatty oils has been made by Holde (The Analyst, xix., 43), who uses ether-alcohol as a solvent for the oil. The author has confirmed Holde's statement that the average amount of matter dissolved from pure rubber by ether-alcohol is 5 per cent., but suggests a slight modification of procedure. Instead of washing the comminuted rubber on the filter with the solvent, the material under examination should be digested with half the total quantity of ether-alcohol (sixty times the amount of rubber taken) in the cold for one hour, filtered, and washed with the remainder of the solvent. The weighing of the residual rubber is also stated to be simpler and quicker than the recovery of the oil. Should surrogate as well as oil be present, the method is not applicable, as the solubility of commercial surrogates in ether-alcohol varies widely. Thus white surrogate gave up 6·96 and 19·65 per cent., brown surrogate 11·05 and 6·69 per cent., castor-oil surrogate 9·85 per cent., and surrogate prepared according to German

patent 73045, 33·96 per cent. to ether-alcohol. It is of course possible that the substance thus extracted from these surrogates may be unaltered oil or its oxidation products which have escaped the action of the sulphur or sulphur chloride used in preparing the surrogate, but the practical result is the same, and discrimination between surrogate (with or without unacted-on oil) and oil added as such is rendered impracticable. On these grounds the author prefers his own methods. A better separation of oil and rubber might be effected by the use of the solvent proposed by Weber (*Zeit. f. angew. Chem.*, 1893, 631), consisting of two volumes of methyl alcohol and one volume of benzene ; but the statement that this mixture dissolves no rubber must be accepted with reservation, the author having found it remove 4·9 per cent. (consisting half of rubber and half of sulphur) from a sample of pure vulcanized Para rubber. No organic solvent with which experiment has been made has been found to be quite without action on rubber.

Estimation of Asphalt and Lampblack in Rubber Goods.—The author alluded to this analytical problem in the earlier part of his investigations, but deferred its solution until the question of determining the commoner admixtures (*e.g.*, surrogate) had been settled. The following are the results at which he has now arrived.

Seeing that both asphalt and rubber are neutral substances remarkably resistant to most chemical agents, separation by means of a suitable solvent is the most promising method of dealing with them. The earlier experiments made in this direction were concerned with the separation of asphalt from unvulcanized rubber. Such organic solvents as chloroform and oil of turpentine, which dissolve asphalt without residue, and convert unvulcanized rubber into a gummy mass difficult to deal with, are therefore unsuitable. Nitro-benzene was found to be a good solvent for asphalt while attacking rubber in minor degree. One gramme of the finely-divided sample is digested in the cold with 30 c.c. of nitro-benzene for one hour, and the whole mass transferred to a filter, where the liquid is expressed as far as possible by means of a small pestle, and the residual rubber washed with a further 30 c.c. of the solvent. The rubber is washed off the filter by a jet of water into a porcelain dish, and the adhering nitro-benzene evaporated, its volatilization being aided by the concomitant water vapour. When all smell of nitro-benzene has ceased, the rubber is again filtered from the unevaporated portion of the water, dried and weighed. Should the rubber contain inorganic constituents which are soluble in water, the last filtration should be avoided, and the residual water evaporated to dryness.

Four samples of rubber were treated in this manner in order to ascertain what loss they suffered. Samples A and B, which were pure rolled raw rubber, lost 1·14 and 2·03 per cent. respectively, while C and D, which were pure "patent" plate rubber, lost 1·10 and 1·54 per cent. A mean correction of 1·5 per cent. must, therefore, be applied in estimating asphalt by this process. The validity of the method was verified by incorporating raw rubber with known quantities of asphalt dissolved in chloroform, drying the whole mass, and extracting it by means of nitro-benzene. The agreement between the found and calculated results was good.

The matter becomes slightly less simple in the case of vulcanized rubber, because of the presence of sulphur therein. The difficulty is overcome by removing the sulphur before determining the asphalt, this object being attained in the course of

the usual treatment with alcoholic alkali to extract surrogate. Provided the alcohol be evaporated before filtering after the alkali extraction, no asphalt is retained in solution. The constant for the loss of vulcanized rubber when treated with nitrobenzene is somewhat higher than that for raw rubber (1·5 per cent.), viz., 3 per cent. Using these precautions and this correction, a good agreement between the calculated and found content of asphalt in vulcanized rubber was obtained by the author. From hard rubber rather less matter is extracted than from soft rubber, but the difference is not great enough to invalidate the correction (3 per cent.) given above. The process is applicable to artificial asphalt made from coal-tar pitch, as well as to natural mineral asphalt. It must be understood that the method needs modification in the presence of mineral oils, paraffin, and similar unsaponifiable substances, which would be unattacked by the alcoholic alkali used to remove fatty oils and surrogate; but cases in which these bodies occur along with asphalt are rare, and can be dealt with as they arise.

Passing to the detection of lampblack in rubber goods, the following considerations serve for the foundation of a method. Experiments by the author confirm the formula $(C_{10}H_{16})_n$ as generally representative of the composition of pure rubber, the limits found being C : H = 10 : 15·31 and C : H = 10 : 17·02. Taking the mean ratio C : H = 10 : 16 as correct, the excess of carbon found by combustion over and above that necessary to provide for the hydrogen represents free carbon, and serves as a measure of the lampblack content. The cardinal failing of this method is that the hydrogen is the basis of calculation, and errors in its determination are largely multiplied in calculating therefrom the carbon present as rubber. The importance of this source of error is minimized in practice by the circumstance that rubber goods containing lampblack are almost certain to contain other adulterants, and an error in the estimation of the lampblack in the residual rubber after removal of surrogate, etc., becomes relatively less considerable when calculated on the original rubber article. (Bearing this in mind, it is obvious that an improvement in closeness of result can be effected by removing the bulk of the rubber before combustion by solution in mineral oil, as suggested by the author in his previous papers, always provided that the fraction of the rubber left undissolved preserves the ratio C : H = 10 : 16.) The author quotes several analyses which prove the substantial accuracy of the process. He concludes the paper by an example of the analysis of a complex specimen of commercial rubber, viz., an india-rubber overshoe. Before describing the actual scheme of analysis, he mentions that he now avoids the separation of the sulphur present as vulcanizing sulphur from that existing as sulphides and sulphates by means of solution of the rubber in petroleum, on the ground that the process is tedious and yields imperfect solution when the rubber is heavily weighted. It is preferable, therefore, to divide the rubber as finely as possible with a rasp, decompose the sulphides by treatment with acid, the H_2S evolved being determined, and estimate the soluble sulphates in the solution thus obtained. The residue can then contain sulphur only in the form of vulcanizing sulphur and insoluble sulphates (*e.g.*, $PbSO_4$ and $BaSO_4$), which are allowed for by calculation from their bases.

The rubber overshoe analyzed was of well-known make, and generally considered

to be of good quality. It was easily reduced to fine powder. One gramme was treated with HCl, and the total gases evolved (CO_2 and H_2S) weighed, the lead sulphide present being completely decomposed. The residue was repeatedly extracted with dilute acid, and the bases determined in the total extract, which was free from sulphate. The total sulphur in the residue was determined by the method previously described by the author, and a trace of lead, which had existed as sulphate (derived from the lead sulphide by accidental oxidation), obtained in the same portion, as well as a little silica and alumina. Another portion of the rubber (1 gramme) was used for the determination of the sulphur in all forms, the difference between this and that in the insoluble residue giving the sulphur evolved as H_2S, and therefore the CO_2 estimated at the same time. A third portion of 5 grammes was treated with acid, filtered, washed, and weighed. Surrogate was looked for in part of the residue, and only a trace found; the small quantity detected may have been nothing but oily matter naturally present in the rubber. Another part of the same residue was used for the determination of asphalt and lampblack in the manner described above. The following are the figures obtained:

Lead oxide	8·45	per cent.
Calcium carbonate	47·81	,,
Ferric oxide and alumina...	1·70	,,
Silica	0·44	,,
Vulcanizing sulphur*	1·50	,,
Oily matter	0·95	,,
Asphalt	8·46	,,
Lampblack	0·39	,,
Caoutchouc	30·30	,,
Total	100·00	

The small percentage of sulphur is noteworthy, as is also the fact that the greater part exists as lead sulphide. As litharge, and not lead sulphide, is used in the manufacture, the function of about one-half appears to be to unite this with sulphur—3·94 per cent. PbO being thus accounted for. The remaining sulphur is only about 2½ per cent. of the caoutchouc, *i.e.*, the quantity absolutely requisite for vulcanization. B. B.

Determination of Chromium in Chromium Steel. J. Spüller and S. Kalman. (*Chem. Zeit.*, 1893, xvii. 1360, 1361 and 1412.)—The authors have extended their method for the determination of chromium in ferrochromium (Analyst xviii., 251) to the determination of the same element in chromium steel.

Two grammes of steel are dissolved in a round-bottomed porcelain basin in 16 c.c. of sulphuric acid (1 : 5 by volume), the solution evaporated to dryness and heated strongly, the crust of sulphates being broken up by means of a glass rod. The dried residue is transferred to a silver dish, intimately mixed with 4 grammes of sodium peroxide and 8 grammes of caustic soda, and the mass gently heated until the evolu-

* Of this 0·61 per cent. was present as PbS, 0·08 per cent. as $PbSO_4$, and 0·81 per cent. as free sulphur and sulphur combined with caoutchouc.

tion of oxygen, which at first takes place from the action of the sulphates on the sodium peroxide, has ceased. The melt should be stirred with a silver spatula during this part of the process to prevent its frothing over. The temperature is then raised and the fusion continued for fifteen minutes, at the end of which time 4 grammes of sodium peroxide are added, followed by another portion of 2 grammes after another twenty minutes. The whole of the chromium is converted into sodium chromate in a time not exceeding one hour and a quarter for the whole series of operations. The silver dish with its contents is allowed to cool until it has reached a temperature of 40° to 50°C, when it is wiped and placed in a roomy porcelain basin and the melt exhausted with water. The sodium manganate and ferrate are removed, as in the case of ferrochromium, by treatment with small successive doses of sodium peroxide, and the excess of sodium peroxide decomposed by passing carbon dioxide through the liquid while hot. The solution of the melt, containing oxides of iron, manganese and aluminium in suspension, is made up to one litre, well-mixed, and an aliquot portion, *e.g.*, 250 c.c. to 500 c.c., according to the richness of the steel in chromium taken for analysis. The rest of the process for the volumetric determination of chromium is conducted as in the analysis of ferrochromium described in the abstract already quoted, save that 25—50 c.c. of sulphuric acid (1 : 5) are used instead of 20 c.c. The mass of sulphates which has to be transferred from the vessel in which the steel is dissolved to that in which the fusion is conducted, is generally easy to detach from the former and leaves but little sticking to the sides. Any traces that may adhere should be wiped out with a small piece of damp filter-paper, incinerated and added to the main quantity. Should the sulphates have been insufficiently dried, the free sulphuric acid accompanying them will decompose much sodium peroxide before the temperature of fusion is reached, so that the addition of a further quantity of the oxidant is necessary.

The method was tested with two samples of chromium steel which nominally contained 1·5 and 4 per cent of chromium. The results were satisfactory, being for the first sample 1·35 and 1·40 per cent., as against 1·43 determined by Blair's gravimetric method (which is usually accepted as accurate though tedious), and 4·08 and 4·10 per cent. instead of 4·10 per cent. by the same check process. The use of the volumetric process described by the authors for determining chromium is not interfered with by the presence of tungsten.

An attempt was made to estimate the amount of chromium in steel directly, without previous solution in acid. It was found that the usual mixture of sodium peroxide and caustic soda was ineffective, and that even sodium peroxide alone failed to attack the metal completely. By hardening the steel, by plunging it while red-hot into cold water, so that it could be broken with the hammer and afterwards reduced to powder in a steel mortar, better results were attained. The subdivision of the metal was best effected by heating the coarse powder obtained as already described, in a Rose's crucible in a stream of oxygen at a low temperature for a period of ten minutes, and rubbing down the oxidized metal in an agate mortar. By treating the product with a mixture of sodium peroxide and caustic soda in the proportion of 10 grammes of the former and 4 grammes of the latter to 2 grammes of the metal and adding another 2 grammes of peroxide during the fusion, complete attack of the

steel can be ensured. The time and labour consumed in comminution are, however, greater than would be required for the solution of the steel in acid.

The use of sodium peroxide is practicable for ferrosilicon and ferrotungsten, and possibly also for ferroaluminium and ferrotitanium. B. B.

The Methods of Testing Fats and Oils. E. Milliau. (*Jour. Franklin Inst.*, 1893, cxxxvi. 376-388 and 433-442.)—This paper contains a description of the methods adopted in the Government Testing Laboratory at Marseilles. The majority of the tests are well known, inasmuch as they differ but slightly from those described in Allen's "Commercial Organic Analysis," vol. ii. The following may be culled, as being of more recent origin:

Examination of Olive-oil for Earth-nut-oil. — Renard's method is modified as follows: 20 grammes of the oil are saponified by 20 c.c. of a caustic soda solution of 36° B., diluted in 100 c.c. alcohol 90 per cent. (*sic*). The soap is precipitated by a 50 per cent. alcoholic solution of neutral lead acetate. The liquid is decanted while warm, and the residue washed with alcohol, ground in a mortar, and agitated with 200 c.c. of ether. The grinding and agitation are repeated, in order to fully extract lead oleate. The residue is then put in a porcelain dish containing 2 or 3 litres of water and 50 c.c. of hydrochloric acid. When decomposition is complete, the solution is decanted, and the fatty acids washed with water, dried in an oven, and dissolved in 40 c.c. of absolute alcohol; a drop of hydrochloric acid is added, and the liquid chilled to 15°. Arachidic acid crystals separate if earth-nut-oil be present.* These are washed first with 90 per cent. alcohol, and then with 70 per cent. alcohol, two lots of 20 c.c. being used in the first case, and three lots of 20 c.c. in the second. The acids are warmed slightly and treated with boiling absolute alcohol. After filtration the alcohol is evaporated, and the residue heated at 100° until constant in weight; it must then melt at 73° to 75°, in order that it may be fully identified as arachidic acid.

In conducting the usual hydrochloric acid and sugar test for sesame-oil in olive-oil the free fatty acids must be used, because the colouring matter of the olive, always present in the oil, gives a reaction with hydrochloric acid and sugar which is indistinguishable from that of sesame-oil.

The author cannot admit Bechi's method for detecting cotton-seed-oil. Instead, he heats 15 c.c. of the oil to about 110°, and pours in, slowly, a mixture of 10 c.c. of a solution of caustic soda of 36° B. with 10 c.c. of alcohol. When the mass boils, 150 c.c. of hot distilled water are added, and the boiling is continued to expel alcohol. The fatty acids are liberated with 10 per cent. sulphuric acid, and immediately collected by a small platinum spoon. They are washed by shaking several times in a test-tube with water; the water is drained off, and the acids poured into a tube 9 cm. long by 2·5 cm. diameter; 15 c.c. of 95 per cent. alcohol and 2 c.c. of a 3 per cent. solution of silver nitrate are added—the tube protected from the light, and heated at 90°, until about one-third of the alcohol is expelled. This is replaced by 10 c.c. of distilled water, and the heating is continued for a few minutes. The black coloration of the fatty acids is now detected if cotton-seed-oil, in any proportion, was originally present.

* Ponzio has recently shown that rape-oil contains about 4 per cent. of arachidic acid.—A. G. B.

For the rapid identification of castor-oil in sesame-oil, 10 grammes are shaken with 4 drops of sulphuric acid (66° B.); a drop of nitric acid (40° B.) is added, and the mixture is shaken violently. Pure sesame-oil blackens immediately, whilst that containing castor-oil remains turbid yellow.

Coco-nut and palm-nut oils are entirely soluble in absolute alcohol; at 30° to 31° the former requires two volumes and the latter four volumes for complete solution. The smallest addition of vegetable or animal oil impairs this solubility. To apply these facts the oil must first be purified by agitating 20 c.c. with 40 c.c. of 95 per cent. alcohol. Oils, such as castor and rosin oil, will be thus detected, for they are soluble in alcohol of this strength; moreover, mowrah and karite oils give a milky turbidity to the alcoholic stratum. Five c.c. of the purified oil are placed in a graduated test-tube, and 10 c.c. of absolute alcohol are added. The temperature is raised to 31°, the tube shaken violently for half a minute, and then immersed in a water-bath kept at a temperature slightly above that of the tube. Pure coco-nut-oil dissolves completely, and the solution remains clear. Any added oil causes precipitation: palm-nut-oil will precipitate from this solution of coco-nut-oil if it amounts to 20 per cent.; otherwise, the mass remains turbid.

Palm-nut-oil is similarly verified, 20 c.c. of absolute alcohol being substituted for 10 c.c. Five c.c. of palm-nut-oil, containing 20 per cent. or more of coco-nut-oil, dissolves in 15 c.c. of absolute alcohol. In the same proportions pure palm-nut-oil does not dissolve, and the mixture remains turbid; a mixture of these two oils, which would appear pure by the iodine absorption and saponification equivalent, would thus be detected by this method. The purity of coco-nut and palm-nut cake may also be determined by first extracting the oil by means of a solvent, and then operating on it in the manner described.

With regard to butter, the author maintains that the determination of the fixed and volatile acids, and of the solubility in alcoholic toluene, will serve to detect admixtures above 10 per cent. Below this point he does not believe that any adulteration would be profitable. A. G. B.

Milk, Skim-Milk, and Whey. By **C. B. Cochran.** (*Amer. Chem. Soc. Jour.*, 1893, xv., 347-351.)—This paper contains some laudatory remarks concerning Richmond's milk scale, which, in the author's opinion, should be authoritatively adopted in the United States, as a check in the analysis of normal milk; chemists who obtain results not in accord with the formula should feel it incumbent on them to investigate the cause of the disagreement.

A number of skim-milks have been analyzed, with a view to ascertaining the specific gravity of casein in solution and the amount by which the specific gravity of the milk is lowered when 1 per cent. of casein is removed. The average of the results shows that each percentage of casein removed lowers the specific gravity of the solution 2·72 "degrees," and that the density of the casein in solution is 1·376.

The figures given for these values, respectively, by Richmond are 2·57° and 1·346; by Dupré, 2·55° and 1·34; by Hehner, 2·36° and 1·3106.

From his analysis of wheys, the author concludes that, when the coagulation is uniformly performed, the specific gravity and total solids of whey vary within

narrow limits, the former varying from 6·5 to 6·9 per cent., and the latter averaging 1·027.

The following table illustrates how a knowledge of the composition and specific gravity of the whey may serve as an aid in determining adulteration by dilution :

No.	Specific Gravity.	Total Solids.	Solids-not-Fat.	Specific Gravity. of Whey.	Total Solids of Whey.
1.	1·0307	14·32	8·78	1·0280	—
2.	1·0315	12·05	8·55	1·0270	—
3.	1·0273	10·90	7·50	1·0230	—
4.	1·0204	12·05	6·15	1·0213	—
5.	1·0318	14·68	9·49	1·0255	5·89
6.	1·0310	12·05	8·45	1·0280	6·59
7.	1·0312	12·45	8·55	1·0270	6·35
8.	1·0320	12·90	8·80	1·0275	6·47
9.	1·0329	13·22	9·02	1·0273	6·42
10.	1·0324	12·98	8·88	1·0283	6·66
11.	1·0329	12·40	8·90	1·0270	6·35
12.	1·0278	8·81	8·71	1·0234	5·50

No. 1. A partially creamed sample of milk.
No. 2. Poor milk.
No. 3. Watered milk.
No. 4. Watered cream.
No. 5. Whey separated after milk had stood three weeks.
No. 12. Watered skim-milk. A. G. B.

REVIEW.

Handbuch der Stickstoffhaltigen Orthocondensations Producte (Handbook of Nitrogenous Ortho-Condensation Products). By O. Kühling. Berlin : Oppenheim. Price 14 marks.

The *raison d'être* of this work shows the marvellous development in Germany of a branch of chemical technology, the origin of which may be traced to the publication of August Kekulé's theoretical views in 1865 on the constitution of benzene, the parent substance of the so-called aromatic compounds.

The subject-matter of this book covers 628 pages. Nitrogenous ortho-condensation products include a great number of the numerous artificially-prepared dyes, and many compounds of therapeutic value.

After a short introduction, the author devotes seventy pages to a general discussion on the constitution, properties, and syntheses of nitrogenous ortho-condensation products. The special part, which follows this, and occupies the remainder of the book, contains for each chapter a theoretical portion and a tabulated list of compounds, showing their constitutions, modes of preparation, and bibliographical references. No mention is made, however, of methods for the detection and estimation of the compounds in question. The rule, adopted by Beilstein in his well-known "Handbuch der Organischen Chemie," of only mentioning compounds of which analytical data have been furnished is carried out as far as possible, but exception is taken in this respect to certain technically-important substances. The technological value of the work is undoubtedly enhanced by references to the patent literature. The author invites his readers to notify errata to him, so that a second edition of the work as free as possible from errors may be prepared. A. R. L.